EM10-OWI-CHM-083

Heat of Combustion
by Parr Bomb Calorimeter 6200

Need
Photo

! This Instruction Contains Descriptions of **HAZARDOUS OPERATIONS** !

Materials and Processes Laboratory
Materials Test Branch, Building 4623

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

Release Authority	Name	Title	Organization	Date
Office of Primary Responsibility	____________________	Materials Test Branch Chief	EM10	_____
	____________________	Industrial Safety	QD50	_____

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Revision	Date	Originator	Description	Affected Pages
Baseline	x/xx/07	Eddie Davis		

This document baselines the Organizational Work Instruction (OWI) for performing Heat of Combustion by Parr Calorimeter 6200 tests in Building 4623. Any deviation to this procedure shall be approved by the test engineer via an approved test plan. Any changes to the test equipment shall be noted on the tester maintenance log and approved by the test engineer. It is the responsibility of the test engineer to obtain NASA Contracting Officer's Technical Representative (COTR) approval where necessary for changes to the test equipment.

Any change to this OWI shall be submitted to and approved by the Materials Test Branch Chief, EM10. Revisions may be also be submitted to the concurring organizations listed below for review and concurrence by memo. The original OWI and all changes shall be maintained by EM10. Any change to materials used requires a change to mechanical drawings, in addition to EM10 Chemistry Team Lead approval. All documentation shall be approved by the appropriate persons mentioned above and incorporated into the OWI before operation of the reconfigured test equipment resumes.

Concurring organizations:
Building 4623 Test Operations Contractor
EM10 Chemistry Team Lead
Environmental Health, AD60M

Contents

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1.0 Scope

1.1 Scope

The scope of this Operational Work Instruction (OWI) is Heat of Combustion by Parr Calorimeter 6200 tests, in accordance with ASTM D240-02, as performed in Marshall Space Flight Center's (MSFC's) Building 4623.

1.2 Purpose

This test method covers the determination of the heat of combustion of materials. The heat of combustion is a measure of the energy available from a fuel. A knowledge of this value is essential when considering the thermal efficiency of equipment for producing either power or heat. The heat of combustion as determined by this test method is designated as one of the chemical and physical requirements of both commercial and military turbine fuels and aviation gasolines. The mass heat of combustion, the heat of combustion per unit mass of fuel, is a critical property of fuels intended for use in weight-limited craft such as airplanes, surface effect vehicles, and hydrofoils. The range of such craft between refueling is a direct function of the heat of combustion and density of the fuel.

The 1104 high-energy oxygen bomb supplied with this instrument is desgined to accommodate routine samples, as well as high-energy fuels and materials. The combustion cage (Figure 9-1) is designed for high-energy samples. The loop electrode configuration (Figure 9-4) is designed for routine samples.

1.3 Applicability

This instruction applies to the Chemistry Team, Materials Test Branch, of the Materials and Processes Laboratory. The test method **shall be used** to measure and describe the properties of materials, products, or assemblies under controlled laboratory conditions and **shall not be used** to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions.

2.0 Applicable Documents

ASTM D240-02. *Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter.*

ASTM D4809-06. *Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method).*

ASTM E144-94. *Standard Practice for Safe Use Of Oxygen Combustion Bombs*

ASTM E711-87. *Standard Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter.*

EM10-OWI-CHM-042. *Test Sample Preparation for Testing in Building 4623.*

EM10-OWI-CHM-050. *Building 4623 Guidelines for Test Operations.*

EM10-OWI-CHM-051. *Receipt, Handling, Prioritizing, and Data Requirements of Samples Submitted for Testing in Building 4623 of the Materials and Processes Laboratory*.

EM10-OWI-CHM-058. *Chemical Hygiene Plan for Building 4623*.

MPD 1840.3. *MSFC Respiratory Protection Program*.

MPR 1040.3. *MSFC Emergency Plan.*

MPR 1840.2. *MSFC Hazard Communication Program*.

MPR 8715.1. *MSFC Safety, Health, and Environmental (SHE) Program.*

MPR 8823.2. *Pressure Systems Guidelines and Certification Requirements.*

MWI 3410.1. *Personnel Certification Program.*

MWI 8621.1. *Close Call and Mishap Reporting and Investigation Program*.

NPR 1600.1. *NASA Security Program Procedural Requirements*

Parr Instruments No. 209M Operating Instructions 1104 Oxygen Combustion Bomb.

Parr Instruments No. 230M Safety in the Operation of Laboratory Reactors and Pressure Vessels.

Parr Instruments No. 442M Operating Instruction Manual 6200 Oxygen Bomb Calorimeter.

Note: Personnel **shall** always **refer** to the current revision of each applicable document.

3.0 Definitions

3.1 Definitions

Energy Equivalent. Effective heat capacity or water equivalent of the calorimeter is the energy required to raise the temperature 1° expressed as MJ/°C: 1 MJ/kg=1000 J/g.

Export Administration Regulations. Regulations set forth in parts 730-774, inclusive, of Title 15 of the Code of Federal Regulations

Gross Heat of Combustion, Qg (MJ/kg). The quantity of energy released when a unit mass of fuel is burned in a constant volume enclosure, with the products being gaseous, other than water that is condensed to the liquid state.

International Traffic in Arms Regulations. Regulations set forth in parts 120-130, inclusive, of Title 22 of the Code of Federal Regulations.

Isoperibol Calorimeter. A calorimeter in which the surrounding jacket is maintained at a constant temperature while the temperature of the bucket and bomb rise as heat is released by the combustion.

Limited access. A term meaning, "Only the test operator shall enter the test cell with appropriate personal protective equipment."

NASA. Marshall Space Flight Center EM10 responsible personnel.

Net Heat of Combustion, Qn (MJ/kg). The quantity of energy released when a unit mass of fuel is burned at constant pressure, with all of the products, including water, being gaseous.

No access. A term meaning, "No one shall enter the test cell."

Proprietary information. Information that is owned by the submitter or representative of the submitter and is viewed by whom the submitter grants access.

Public information. Information accessible to the interested public in any form.

Sensitive But Unclassified. In NPR 1600.1, *NASA Security Program Procedural Requirements*, paragraph 5.24 of Chapter 5.

Tag out. Placement of a tag-out device on an energy-isolating device to indicate that the energy-isolating device and equipment being controlled shall not be operated until the tag-out device is removed by the person who placed it there.

Test area. Building 4623, Room 134

Test engineer. The person responsible for correctly following the approved test plan for a specific test -- from sample receipt to test data evaluation.

Test operator. The person responsible for conducting a test under the guidance of the test engineer.

3.2 Acronyms

EAR	Export Administration Regulations
ITAR	International Traffic in Arms Regulations
MSDS	Material Safety Data Sheet
MSFC	Marshall Space Flight Center
OWI	Organizational Work Instruction
SBU	Sensitive but Unclassified

4.0 Instructions

All operations of this equipment **shall be conducted** using the applicable documents referenced above (section 2). All data and test results **shall be recorded** on the Heat of Combustion by Parr Bomb Calorimeter 6200 data sheet (section 7, Figure 7-2). A summary of pertinent test information and test results **shall be compiled** in an NASA memo, **signed** by the test organization management, and **mailed** to the test requester.

4.1 Sample Preparation

The *sample preparation technician* **shall prepare** test samples according to EM10-OWI-042, *Test Sample Preparation for Testing in Building 4623.* When non-standard samples are to be tested, the *sample preparation technician* **shall follow** the directions written in the test plan for that test request. *If this information is not provided with the test plan*, the *sample preparation technician* **shall seek clarification** from the test engineer.

The sample boats are 1 in. in diameter x 7/16 in. deep. The sample is cut so that it will fit into the sample boats. **Consult** the sample Material Safety Data Sheets (MSDSs) for information regarding the estimated heat of combustion of the sample. The optimal heat of combustion is close to that of the benzoic acid standard (6318 cal/g). *If the sample is an unknown, it* is recommended that 0.4-0.5 g be tested initially for an approximation of the heat of combustion for that material. *If the heat of combustion is very low,* it may be necessary to spike the samples with mineral oil. (**See** section 7.4.)

Before testing begins, the *test operator* **shall review** the information supplied on the test data sheet (prepared by the sample preparation technician) to make certain the information is complete and appears sound. *If a problem is identified*, the *test operator* **shall notify** the test engineer. The *test operator* **shall** also:

- **Note** whether the test folder is marked Sensitive But Unclassified (SBU). *If so,* the test folder, samples, and test data **shall be handled** in accordance with NPR 1600.1, *NASA Security Program Procedural Requirements*, paragraph 5.24 of Chapter 5. Such handling **shall include** storage, access, disclosure, protection, transmittal, and destruction of SBU.
- **Verify** that the test request number and material designation are identical on all paperwork.
- **Confirm** that the prepared samples agree with the test request.
- **Verify** that the sample preparation technician has noted if the sample has been cleaned or if the sample does not require cleaning.
- **Note** any flaws or imperfections in the sample, and **record** these on the test data sheet.

- **Review** the test plan signed by NASA and the original test request before proceeding. *If the test plan and the test request do not agree*, **request** clarification from the test engineer, who shall query NASA.
- All pertinent information for the test, such as sample identification and pretest information about the sample, **shall be recorded.**

4.1.1. Particle Size and Moisture Content

Solid samples burn best in an oxygen bomb when reduced to 60 mesh (or smaller) and compressed into a pellet using a pellet press. Large particles may not burn completely, and small particles are easily swept out of the capsule by turbulent gases during rapid combustion.

Materials like coal burn well in the as-received or air-dried condition; however, **do not burn** completely dry samples. Some moisture is desirable to control the burn rate. Moisture content up to 20% can be tolerated in many cases, but the optimum moisture is best determined by trial combustions. *If moisture is to be added to retard combustion,* **drop** water directly into loose samples or a pellet after the sample has been weighed. **Let** the sample stand awhile to obtain uniform moisture distribution.

Incomplete combustion is indicated by the presence of excess residue (a very slight amount of residue is acceptable) that remains in the combustion capsule after the test. The following paragraphs describe techniques to assist in combustion in these types of samples.

Note: Particle size is important because it influences the reaction rate. Compression into a pellet is recommended because the pressure developed during combustion can be reduced as much as 40% compared to the combustion of the material in powder form. In addition to controlled burning, pelletizing keeps the sample in the fuel capsule during combustion.

4.1.2. Combustion Capsules

Non-volatile samples to be tested in Parr oxygen bombs are weighed and tested in shallow capsules measuring 1 in. (dia.) by 7/16 in. (deep). Stainless-steel capsules are provided with each calorimeter. When combusting samples containing magnesium or aluminum, fused silica capsules are required.

It is recommended that the capsules be heated at 480 °C for 4 hr in a muffle furnace before use. This firing protects the capsule and assists in burning the last traces of a sample. **Repeat** this treatment after the capsule has been polished with an abrasive material to remove leftover surface deposits. This treatment is also a good way to burn off any remaining carbon from previous samples.

Note: Always **handle** the capsules with forceps only, and **place** in a clean container between uses.

4.1.3. Combustion Aids: Some samples may be difficult to ignite, or they may burn so slowly that the particles become chilled below the ignition point before complete combustion occurs. In such cases, powdered benzoic acid can be mixed with the sample; however, this adds to the overall energy released, and the amount of sample may have to be reduced to compensate for this added charge. When benzoic acid is added to a sample, always **form** the sample into a pellet to avoid damage to the bomb.

4.1.4. Sample Types

4.1.4.1. Foodstuff and Cellulosic Materials: Fibrous and fluffy materials generally require one of three modes of controlling the burn rate. Fibrous materials cannot be formed into pellets readily and generally require moisture content from a combustion aid such as mineral oil to retard the burn and avoid development of high pressures. Partial drying may be necessary if moisture content is too high to obtain ignition, but *if the sample is heat sensitive and cannot be dried,* a water soluble combustion aid such as ethylene glycol can be added to promote ignition.

4.1.4.2. Coarse Samples: In most cases, it may be necessary to burn coarse samples without size reduction since grinding or drying may introduce unwanted changes. This is acceptable *if the coarse sample will ignite and burn completely.* Whole wheat grains and coarse charcoal chunks are typical of materials that will burn satisfactorily without grinding and without additives or a special procedure.

4.1.4.3. Corrosive Samples: *If unusually corrosive samples are to be tested,* use of the 1108CL bomb is strongly recommended. The 1108CL bomb is made specifically for such samples. This bomb resists the corrosive effects of high chlorine content.

4.1.4.4. Explosives and High Energy Fuels: The 1104 high-pressure oxygen bomb is designed specifically to test these types of samples. The 1108 bombs are designed for routine samples. See Safety Precautions (step 6.2.6) for advice on working with true explosives.

4.1.4.5. Volatile Samples: Volatile samples can be handled in a Parr 43A6 Platinum Capsule or a Parr 43AS Alloy Capsule. These holders can be sealed with a disc of plastic adhesive tape stretched across the rim of the cup, with the excess tape trimmed away. This will be adequate for most volatile samples. This tape should have a low chlorine and sulfur content. For more information about volatile samples and holders, **see** the 6200 Calorimeter Operating Instruction Manual.

4.1.5. *If only partial combustion was possible,* it may be necessary to have additional samples prepared. The test engineer **shall make** this decision.

4.2 Pre-Test Photography

The *sample preparation technician* **shall take** a pre-test photograph of at least one of the samples and **place** three copies of the photograph in the test folder. *If the pre-test photograph has not been taken*, the *test operator* **shall take** the photograph and **place** three copies of the photograph in the test folder before conducting the test. The entire sample **shall be visible** in the photo. Steps for sample photography are outlined in the *Photography Operating Guide*.

If complete combustion was not possible, the test technician **shall photograph** one representative sample of the residue remaining after the test.

4.3 Equipment Checkout

At the beginning of the test day, the *test operator* **shall perform** the following steps:

4.3.1. **Ensure** that the test system is clean and that all measuring devices are in current calibration.

4.3.2. **Ensure** that there is a light coating of oxygen-compatible antiseize lubricant on the threads of the cap, but **do not use** this or any other lubricant on any other parts of the bomb.

4.3.3. **Verify** proper instrument operation by running a benzoic acid pellet as a standard.

4.3.4. **Ensure** that the water handling system maintains a constant temperature from 24 to 27 °C (75.2 to 80.6 °F).

4.3.5. **Ensure** that the K bottle has at least 450 psi of oxygen available.

4.4 System Setup and Calibration

The *test operator* **shall perform** the following steps for initial system setup:

4.4.1. **Fill** the water reservoir of the calorimeter with approximately 1.4 liters of water (distilled or de-ionized). **Perform** filling before applying power to the calorimeter. **Fill** the reservoir through the tank fill elbow on the back of the calorimeter. The tank is full when water stands in the horizontal run of the filling elbow.

4.4.2. The water handling system (6510) is designed to provide a means to refill the water bucket between tests, as well as to provide a constant water temperature for determinations. The temperature is maintained between 24 and 27 °C (75.2 to 80.6 °F). About 3 hr are required for the system to warm up from a cold start [20 °C (68 °F)] to a constant temperature. For this reason, it is recommended that the water handling system be turned on early in the morning or the night before and be left on the entire week that testing is performed, until testing is completed.

4.4.3. **Turn** the power switch to the **ON** position. After a short time, the Parr logo will appear on the LCD display followed by a running description of the instrument boot sequence. When the boot sequence is complete, the calorimeter Main Menu is displayed. **Go** to the Calorimeter Operation page and **turn** the heater and pump on to begin circulating and heating the calorimeter jacket water. **Add** water to the filling elbow at the rear of the instrument as required to keep it full.

4.4.4. **Determine** the energy equivalent of the calorimeter by averaging not less than six tests using standard benzoic acid (C_6H_6tCOOH) pellets. These tests should be spaced over a period of not less than 3 days. **Use** not less than 0.9 g nor more than 1.1 g of standard benzoic acid. **Make** each determination according to the procedure described in section 4.5, and **compute** the corrected temperature rise (t). (**See** section 7.X for additional information.)

4.4.5. **Perform** the standardization by following steps 4.5.2 through 4.5.16 in Detailed Test Procedures (below).

4.5 Detailed Test Procedures

The test operator **shall perform** the following steps:

4.5.1. **Turn on** the calorimeter using the switch on the lower left hand side in back.

4.5.2. **Turn on** the water handling system located at the lower left hand side in back. The system takes a long time (3 to 5 hr) to heat up from room temperature and has to maintain an accurate temp of 26 to 28 °C (78.8 to 82.4 °F). **Turn on** the water handler the night before if running samples the next day, and **ensure** there are no water leaks. *If a number of samples are to be run over several days,* **leave** the water handler on until finished with that group of samples. A container of microwaved water may be used to speed up heating. **Be sure** to use deionized or distilled water.

4.5.3. At the calorimeter touch screen, go to **Calorimeter Operations**, and **click on Heater and Pump**. The water takes 20 to 30 min to warm to 30 °C (86 °F). Once the jacket temperature reaches 30 °C (86 °F) and remains there for 10 minutes, the calorimeter is ready for testing. After 20 to 30 minutes the *Start* button on the **Calorimeter Operations** screen should be lit, indicating the system is ready for testing.

4.5.4. **Review** the MSDS for the sample, and **fill out** the Pre-Test Checklist (Figure 7-1).

4.5.5. **Wet** the top part of the calorimeter sealing surface, and gently **shake off** the residual water. This serves to wet the sealing parts of the bomb and leaves the same amount of residual water as will remain in subsequent testing.

4.5.6. **Run** one or two benzoic acid runs as samples, then possibly one as a standard. **Choose Determinations** or **Standardizations** on the **Operating Mode** button of the **Calorimeter Operations** screen. **Choose Determinations** for the first run and **Standardization** for the second if adding this in the standards set of statistics data. Otherwise, **run** the second standard as **Determinations** as well.

Note: Benzoic acid is a good standardization material because it is stable, not hygroscopic, can be easily pressed into a pellet, and has a calorific content in the range of many common samples. The benzoic acid pellets are provided already prepared as 1-g pellets with the bomb calorimeter. The energy value for the benzoic acid should be around 6318 cal/g.

Note: To print out all standardization data, **go** to **Calibration Data and Control** from the main menu. **Press Escape** to go to the previous screen.

4.5.7. For the lower energy samples (samples that do not use the cage), **select 1/2517.8** on the **Bomb/EE** button of the **Calorimeter Operations** screen for the bomb identification. For the higher energy samples (those that use the combustion cage) or for unknowns, select **2/2547** on the **Bomb/EE** button.

4.5.8. **Open** the bomb compartment, and **remove** the bucket. **Move** the firing wires to the side. **Fill** the bucket from the auto-filling pipet by turning the stopcock 180 degrees to fill it slightly. **Add** about 1 ml of deionized water from the autopipet to the bottom of the bomb. This water should only have to be added to the bomb once a day until it is cleaned at the end of the day. **Completely refill** the autopipet by turning the stopcock 180 degrees again. The autopipet fills and drains continually and automatically so that a constant volume of 1850 ml (±1.5 ml) of water is maintained. **Add** this entire amount to the bucket each time it is filled for a run.

4.5.9. **Open** the stem valve on the oxygen K bottle very slowly. **Adjust** the pressure regulator until it reads 420 psi.

4.5.10. **Prepare** the standard bomb sample holder. With the sample holder in its ring stand, **cut** 10 to 12 cm (3.9 to 4.7 in.) of firing wire with the scissors. **Run** the wire through the holes like threading a needle. **Run** the wire through the side without a hook slightly and bend it around the post one turn above the hole. Below the hole, **run** the wire around the post two more times to make sure it is secure. **Run** the other end of the wire to the other post with the hook. **Run**

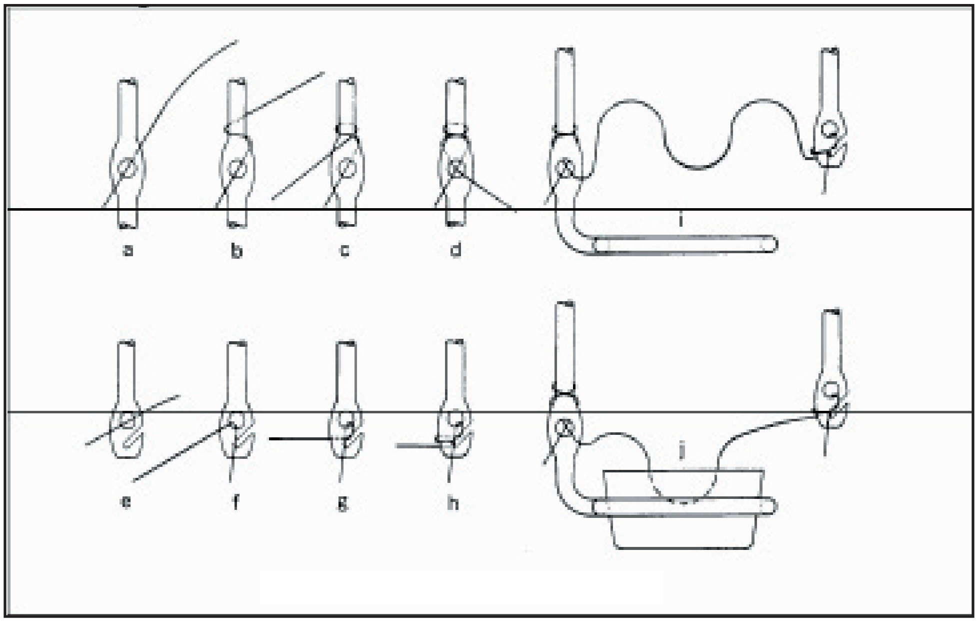


Figure 4.5-1.
Steps for connecting fuse wire to electrodes

the wire through the hole and one turn around above the hole. Below the hole, run the wire around the hook twice more to secure it. (See Figure 4.5.1 for the correct firing wire connection procedure.) **Weigh** the sample on the analytical, and **record** the weight. **Place** the sample in the sample holding cup. **Go** to step 4.5.13 to continue to load the standard energy sample holder.

4.5.11. For the high-energy sample holder, the process is somewhat more difficult. **Invert** the sample holder so it is upside down in the ring stand. **Remove** the standard sample holder by unscrewing it from the hole at the sample holder. **Screw** in the high-energy sample holder until it is finger tight. **Cut** 12 to 15 cm (4.7 to 5.9 in.). of firing wire. **Hook** one end of the wire under the screw on the outside of the cage. **Tighten** the screw securely. **Run** the other end of the wire through the hole closest to the screw. **Thread** the wire through the cage and out through the open portion in the bottom of the cage. The end that is not attached will attach to same hook as that mentioned in step 4.5.10. **Run** the wire around the hook twice and ensure it is securely fastened. **Loosen** the wire at the other end that is attached to the screw, and **pull** it out slightly so it does not touch the sides of the hole. **Turn** the sample holder over so it is in the proper orientation for loading the sample.

CAUTION: The wire should not touch the cage except at the ends, otherwise the wire will short out and a misfire will occur. **Use** a light to verify that the wire is not touching the sides of the hold.

Note: *If misfire message appears in the middle of a run,* this indicates that no temperature change was detected and that the wire did not fire. **Remove** the bomb, **bleed** it down, and determine the cause. This is much more common with the high-energy sample holder than the standard energy sample holder.

Note: This procedure is easier if the wire is not kinked more than necessary. **Use** tweezers to pull the wire through the cage, and **bend** it in the proper directions.

4.5.12. Either **tare** the sample holder cup and **weigh** the sample in it, or **weigh** the sample and **place** it in the sample cup already in the ring on the sample holder. **Record** the sample weight. For the standard sample holder, simply **place** the cup securely in the holder. **Tighten** the screws for the high-energy sample cage. Bend the wire so that it is suspended slightly above the sample or touching **the** sample.

CAUTION: It is critical that the wire only touch the metal of the sample holder on the two ends of the wire. A misfire may result.

4.5.13. **Place** the sample holder in the bomb until the o-ring seats against the bottom half of the bomb. **Ensure** the compression ring is in place above the head of the bomb. **Screw** the top half of the bomb to the bottom half until it is finger tight. **Tighten** the Allen screws in a star pattern with the Allen wrench until they are all just past finger tight. *If one screw tightens significantly more or less than the others,* **loosen** all the screws and **begin** tightening them again, else the screws will not be tightened equally.

4.5.14. **Fill** the bomb with oxygen. Removed the larger knurled thumb screw from the bomb. Ensure that the outlet vent is closed finger tight. Attach the oxygen line to the connection finger tight. **Press O2 Fill** on the **Calorimeter Operations** screen to fill the vessel. (It take approximately 60 sec to fill the bomb.) At the end of the normal fill cycle, the valve will vent off. **Remove** the oxygen fill line and **replace** the knurled screw.

Note: *If a leak is evident (indicated by hissing,*) **press** the **O2 Fill** button again to abort the filling operation, and **vent** the bomb by loosening the vent valve. **Verify** that all the screws are tightened.

Note: The default filling pressure is set to 420 psi. The maximum pressure allowable is 600 psi. *If a pressure other than 420 psi is planned,* the instrument must be recalibrated with the benzoic acid (see section 4.4) at this same pressure.

4.5.15. Using the bomb lifter, **raise** the bomb into the bucket carefully until it mates with the raised portion in the center of the bucket. **Put** the filled bucket back in the bomb compartment so it mates with the three raised grooves. **Ensure** there are no leaks (indicated by a significant number of air bubbles; a few bubbles are normal, indicating air was released below the bomb when it is set into place).

CAUTION: If there are leaks, **DO NOT continue** the test. **Remove** the bomb from the bucket, bleed down the bomb, and fix the leak.

4.5.16. **Attach** the two firing igniter wires to the bomb. These slip on and it does not matter to which plug they are attached. **Ensure** the wires are out of the way of the stirrer, and **close** the cover carefully. **Close** the lid of the calorimeter.

4.5.17. Press the **START** button on the **Calorimeter Operations** screen. If this button is not lit, this indicates that the water handler is probalby not at temperature [25 to 29 °C (77 to 84.2 °F)] or the bucket and its pump are not at temperature [30 °C (86 °F). By default, the reporting units are calories/gram (cal/g). Reporting units can be changed at the main screen by selecting **Operating Controls** and then **Reporting Units**.

4.5.18. **Select** **Determination** or **Standardization** as appropriate on the **Calorimeter Operations** screen. **Change** the default name on the **Auto Accept Sample ID** screen by pressing **NO** and entering a different name. **Use** the following format for the file name: item number-sample run number, *e.g.,* 105123-1. The bomb ID should be "1" for regular energy samples and "2" for the high-energy sample holder. **Enter** the sample weight recorded in steps 4.5.10 or 4.5.12.

4.5.19. At this point, the run begins. The **PREPERIOD** message indicates the time before firing while the instrument waits for temperatures to stabilize. The full run time is 15 min, but it often takes less time. The instrument waits until the temperatures are both stable and then fires the sample. Four short beeps indicate the instrument is about to fire.

CAUTION: *If running an unknown,* **stand back** from the instrument at this point.

The instrument waits for the bucket temperature to rise and waits for the rise to end, and the run is complete. The time when the instrument is waiting for the temperatures to stabilize after the test is indicated by **POSTPERIOD.** The printer automatically prints the results at the end of each run.

4.5.20. **Open** the lid, and **remove** the firing wires from the bomb.

4.5.21. **Remove** the bomb, and **pour** the water on the top of the bomb back into the bucket. **Place** the bomb on the ring stand. **Pour** the water from the bucket slowly back into the water handling system. **Dry** the bottom of the bucket. **Refill** the bucket from the autopipet each time to ensure the same amount of water is used for each run (1850 ml).

4.5.22. **Vent** the oxygen from the bomb after the run by loosening the smaller knurled nut slowly to release the oxygen slowly. *If gases being released are or may be toxic,* **vent** the bomb under a fume hood. **Loosen** the Allen screws in the same fashion as tightened initially. *If the Allen screws are very hard to turn,* the bomb has not been vented, is not completely vented to atmosphere, or the Allen screws were not loosened properly. **Follow** the same procedure for loosening the screws as for tightening them. It may be necessary to retighten the screws and then loosen them in the proper order.

Remove the top half of the bomb after the bomb reaches atmospheric pressure, and **examine** the sample cup. Incomplete combustion is indicated by excessive residue in the cup or the bottom of the bomb. If this occurs, **consider** adding mineral oil or benzoic acid as a promoter. (**See** section 7.X.) **Remove** any unburned wire from the two ends attached to the sample holder.

Note: *If incomplete combustion is indicated or the energy indicated is very low,* **use** mineral oil as a promoter or combustion aid. (**See** sections 7.3 and 7.4 for this procedure.)

4.5.23. Repeat steps 4.5.10 through 4.5.22 to perform another run. Typically, each sample is run at least three times to obtain acceptable good data.

Note: The bomb calorimeter is an extremely precise instrument. Large fluctuations among multiple runs of the same sample probably indicate incomplete combustion or improper sample preparation. The benzoic acid standardization should be very close to the published values, probably within 1%. It is suggested that initially each sample be run in triplicate to validate instrument accuracy and precision.

4.5.24. **Separate** the printouts for each item number, and **place** them in the proper folder. **Record** the data on the Heat of Combustion Test Data Sheet (Figure 7-2).

Note: To check previous data, **go** to the **Main Menu** of the calorimeter touch screen. **Click** on **File Management**; then click on **Run Data File Manager**. To reprint data, touch **Display** and then **Print**. Files can be renamed, deleted, and reprinted from this screen.

4.5.25. At the end of each testing day, **dry** the top half of the bomb and the inside of the bomb. **Ensure** that no tissue remains in the bomb after drying. **Dry** the top of the sample holder. **Turn off** the calorimeter. **Close** the oxygen K-bottle stem valve. **Turn off** the regulator until it is fully closed.

5.0 Notes

Custodians for EM10-OWI-CHM-083	
Master List and Document Control	EM10 Management Support Assistant
Alternate Document Control	EM10 Group ISO Representative
Memoranda	Materials Test Branch ISO Representative

6.0 Safety Precautions and Warning Notes

6.1 Hazards

Warning
Death, severe personal injury, or loss of major equipment may result if maintenance or operating procedures, techniques, restrictions, etc., are not followed exactly.

Safety shall have precedence over all activities. Because of the nature of testing with flammable materials, the testing system involves several hazards to the operator and facility. These include:

- Potential touch temperature risks from hot surfaces
- Burning materials in air or oxygen-enriched environments
- Pressurized nitrogen and oxygen supply systems
- Electrical load and other ignition sources applied in air, oxygen-enriched environments, and combustible by-products
- Flammable and dangerous liquid solvents
- Electrical valves, power supplies, switches, and other components
- Oxygen deficiency.

6.2 Safety Precautions

Personnel **shall:**

6.2.1. **Plan** test setup, testing, and shutdown so that at least one test operator is in the test area and one other person is in Building 4623 during normal business hours. After normal business hours and on weekends, a test engineer shall be in Building 4623 during all test activities. **No more than five people** shall be in the test area at any given time. Operation of tests shall comply with EM10-OWI-CHM-050, *Building 4623 Guidelines for Test Operations.*

6.2.2. **Refer to the MSDS** for information on personal protective equipment required required for handling sample materials, solvents, gaseous nitrogen, and gaseous oxygen. **Wear**:

- Safety shoes when there is a danger of foot injuries from falling or rolling objects, objects piercing the sole of the shoe, or when feet may be exposed to an electrical hazard
- Clean laboratory coat when working with enriched oxygen or other oxidizers, combustion by-products, compressed gases, or flammable solvents
- Safety glasses at all times while in the test cell
- Chemical goggles and gloves while cleaning test equipment and while working with solvents.
- A respirator when working with solvents in closed or poorly ventilated spaces. **Note** that the appropriate respirator shall be worn as indicated on the solvent's MSDS. Cartridge respirators are only good for the constituents listed on the filtration cartridge and for dust particle filtration. Supplied air respirators shall be mandatory for exposure to certain chemicals. Personnel shall be qualified to use the respirator, and the respirator shall be supplied by MSFC.

6.2.3. **Smoking is not permitted** in Building 4623. The test area is generally an oxygen-enriched environment. Open flame or other high-temperature sources are not permissible in the testing area while enriched-oxygen conditions exist. **Do not smoke or expose clothing** to an open flame for 30 minutes after handling liquid or gaseous oxygen.

6.2.4. **Activate** the building warning system for the duration of all testing, including pre- and post-test procedures. **Evacuate** the test area immediately when the oxygen alarm sounds and lights flash.

6.2.5. When making connections for compressed gases, **refer** to *Working Safely with Compressed Gases and Cryogens* and *NSTC 313-Cryogenics Safety.* (**See** the test engineer for these resources.) **Comply** with the suggestions inside these presentations.

6.2.6. It is not advisable to test true explosives in the presence of oxygen. Some of the end products of the explosion are combustible in high-pressure oxygen, *e.g.,* mixed oxides of nitrogen and carbon monoxide. With high-pressure oxygen available, these burn to carbon dioxide and release an undeterminable amount of heat that did not come from the initial reaction of interest. This secondary combustion can give off nearly as much energy as the preliminary explosion. To eliminate this concern, **fill** the bomb with nitrogen, and then **release** the valve to flush the vessel; **repeat** this process twice to reduce the atmospheric oxygen trapped in the bomb to a level that is insignificant, relative to the primary reaction. **Leave** a residual pressure of at least 5 atmospheres on the bomb to seat the inlet check valve.

6.2.7. **Check** building warning lights for proper operation before testing begins.

6.3 Special Hazards Associated with Compressed Gases and Liquids

6.3.1. All operations involving compressed gases and liquids shall be conducted with at least 2 people, in visual contact, in the facility.

6.3.2. All operating personnel shall be instructed on the nature of hazards associated with compressed gases and liquids.

6.3.3. Before removal of any component of the system for servicing, the *operator* **shall secure and inspect** the system to verify that no unsafe condition exists.

6.3.4. Personnel shall perform continuous monitoring, *e.g.,* check operating pressures, look for leaks, listen for unusual noises, during all operations. Personnel shall verify that oxygen leak levels are at or near zero throughout operations.

6.4 Emergency Shutdown Procedure

The tester does not have to be shut down to be considered safe in an emergency situation.

6.5 Accident Reporting

6.5.1. From a safe location, the *test operator* **shall call 911 immediately** and **notify** the EM10 Materials Test Branch Chief.

6.5.2. From a safe location, the *EM10 Materials Test Branch Chief* **shall immediately report** the accident to the NASA Safety Monitor and the appropriate supervisor(s).

6.6 Emergency Response Plan

Emergency procedures and plans for Building 4623 are incorporated into this OWI and are stated in MPR 1040.3, *MSFC Emergency Plan.* Plans shall be modified if operations change in a significant manner.

6.7 Mishap Reporting

Personnel **shall report** all mishaps occurring in Building 4623 to the test engineer, who shall report the mishap to the area coordinator/Safety Monitor. An initial verbal report shall be made within 8 hours, followed by a written report within 3 days. The EM10 Chemistry Team Lead shall prepare a managerial report within 7 days. Both reports shall be reviewed by the test operator's supervisor and by the NASA Safety Monitor. The detail and extent of the mishap report shall depend on the nature and extent of the damage. *If personnel injury or equipment damage does occur*, the mishap report shall be completed in accordance with MWI 8621.1A, *Close Call and Mishap Reporting and Investigation Program*.

7.0 Attachments, Data, Reports, and Forms

7.1 Attachments

7.1.1. Poor Overall Combustion of Sample

Poor combustion is indicated by the presence of soot or unburned sample following the combustion test. *If these are present,* **discard** the test results, and **test** again. Because of the difference in combustion characteristics of the many different materials that may be burned in an oxygen bomb, it is difficult to give specific directions that will assure complete combustion of all samples.

Consider the following factors when performing bomb calorimeter testing:

1. Some part of the sample must be heated to its ignition temperature to start the combustion. In burning, the sample must liberate sufficient heat to support its own combustion regardless of the chilling effect of adjacent metal parts.
2. The combustion must produce sufficient turbulence within the bomb to bring oxygen into the fuel cup for burning the last traces of the sample.
3. Loose or powdery samples may cause unburned particles to be ejected during a violent combustion.
4. The use of a sample that contains coarse particles will not burn readily. Coal particles that are too large to pass through a 60-mesh screen may not burn completely.
5. The sample pellet may be too hard of too soft. Either condition can cause spalling and the ejection of unburned fragments.
6. Space between the combustion cup and the bottom of the bomb may be insufficient. The bottom of the cup should always be at least 1/2 in. above the bottom of the bomb or above the liquid level in the bomb to prevent thermal quenching.
7. The sample may contain excessive moisture or non-combustible material. *If the moisture, ash, and other noncombustible material in the sample amounts to 20% or more of the charge,* it may be difficult to obtain complete combustion. This can be remedied by adding a small amount of benzoic acid to the sample.

7.1.2. Standardization Procedure

The term standardization denotes the operation of the calorimeter on a standard sample from which the energy equivalent or effective heat capacity of the system can be determined. The energy equivalent, W or EE, of the calorimeter is the energy required to raise the temperature 1 degree, usually expressed as calories per °C. Standardization tests should be repeated after changing any parts of the calorimeter and occasionally as a check on both thecalorimeter and operating technique.

The procedure for a standardization test is exactly the same as for testing a fuel sample. **Use** a pellet of calorific grade benzoicacid weighing not less than 0.9 nor more than 1.25 g. The corrected temperature rise, T, is determined from the observed test data and the bomb washings are titrated to determine the nitric acid correction. The energy equivalent is computed by substituting the following equation:

$$W = (Hm + e_1 + e_2 + e_3)/T \qquad \text{(Eq. 1)}$$

where:

W = Energy equivalent of the calorimeter (calories/° C)
H = Heat of combustion of the standard benzoic acid sample (calories/g)
m = Mass of the standard benzoic acid sample (g)
T = Temperature rise (°C)
e_1 = Correction for heat of formation of nitric acid (calories)
e_2 = Correction for sulfur (usually 0)
e_3 = Correction for heating wire and combustion of cotton thread.

Take care to ensure that the conditions during standardization runs and determinations are as identical as possible.

7.1.3. Calculations (performed by bomb calorimeter).

The following is provided for information only. The bomb calorimeter accurately determines these values.

7.1.3.1. Temperature Rise in Isothermal Jacket Calorimeter: Using data obtained as prescribed in section 4.5, the temperature rise (t) in an isothermal jacket calorimeter is computed as follows:

$$t = t_c - t_a - r_1(b - a) - r_2(c - b) \qquad \text{(Eq. 2)}$$

where:

t = corrected temperature rise
a = time of firing
b = time (to nearest 0.1 min) when the temperature rise reaches 60% of total
c = time at beginning of period in which the rate of temperature change with time has become constant (after combustion)
t_a = temperature at time of firing, corrected for thermometer error
t_c = temperature at time (c) corrected for thermometer error
r_1 = rate (temperature units per minute) at which temperature was rising during 5-min period before firing
r_2 = rate (temperature units per minute) at which temperature was rising during the 5-min period after time c. *If the temperature is falling,* r_2 is negative and the quantity $-r_2(c - b)$ is positive.

7.1.3.2. Temperature Rise in Adiabatic Jacket Calorimeter: Using data obtained as prescribed in 4.5, the temperature rise (t) in an adiabatic jacket calorimeter is computed as follows:

$$t = t_f - t_a \qquad \text{(Eq. 3)}$$

where:
t = corrected temperature rise
t_a = temperature when charge was fired, corrected for thermometer error
t_f = final equilibrium temperature, corrected for the thermometer error.

7.1.3.3. Thermochemical Corrections. Compute the following for each test:

e_1 = correction for heat of formation of nitric acid (HNO_3); MJ = cm^3 of standard (0.0866 N) NaOH solution used in titration X $5/10^6$
e_2 = correction for heat of formation of sulfuric acid (H_2SO_4); MJ = 58.0 X percentage of sulfur in sample X mass of sample/10^6
e_3 = correction for heat of combustion of firing wire (MJ)
= 1.13 3 mm of iron wire consumed/10^6
= 0.96 3 mms of Chromel C wire consumed/10^6
e4 = correction for heat of combustion of pressure-sensitive tape or gelatin capsule and mineral oil; MJ = mass of tape or capsule oil, g X heat of combustion of tape or capsule/oil (MJ/kg/$10^{6)}$.

7.1.3.4. Gross Heat of Combustion: the gross heat of combustion is computed by substituting in the following equation:

$$Q_g = (tW - e_1 - e_2 - e_3 - e_4)/1000\ g \qquad \text{(Eq. 4)}$$

where:
Q_g = gross heat of combustion, at constant volume expressed as MJ/kg
t = corrected temperature rise (steps 4.6.41 or 4.6.4.2) (°C)
W = energy equivalent of calorimeter (MJ/°C) (step 4.4.1)
e_1, e_2, e_3, e_4 = corrections as prescribed in step 4.8.4.3
g = weight of sample (g).

Note: The gross heat of combustion at constant pressure may be calculated as follows:

$$Q_{gp} = Q_g + 0.006145H \qquad \text{(Eq. 5)}$$

where:
Q_{gp} = gross heat of combustion at constant pressure (MJ/kg)
H = hydrogen content (mass %).

7.1.3.5. Net Heat of Combustion: The net heat of combustion is represented by the symbol Q_n and is related to the gross heat of combustion by the following equation:

$$Q_n\ (\text{net}, 25\ °C) = Q_g\ (\text{gross}, 25°C) - 0.2122 \times H \qquad \text{(Eq. 6)}$$

where:
Q_n (net, 25°C) = net heat of combustion at constant pressure (MJ/kg)
Q_g (gross, 25°C) = gross heat of combustion at constant volume (MJ/kg)
H = mass% of hydrogen in the sample.

7.1.3.5.1. *If the percentage of hydrogen, H, in the sample is known,* the net heat of combustion may be calculated from Eq. 6.

When the percentage of hydrogen is not known, **determine** the hydrogen in accordance with Test Methods D1018.

7.1.3.5.2. *If the percentage of hydrogen in aviation gasoline and turbine fuel samples is not known,* the net heat of combustion may be calculated as follows:

$$Q_n = 10.025 + (0.7195)Q_g \qquad \text{(Eq. 7)}$$

where:
Q_n = net heat of combustion at constant pressure (MJ/kg)
Q_g = gross heat of combustion at constant volume (MJ/kg).

Note: Eq. 7 is recommended only if the percentage of hydrogen is not known. It is based on Eq. 6 and an empirical relation between Q_n and the percentage of hydrogen in aviation gasolines and turbine fuels.

7.1.3.5.3. Net heat of combustion is the quantity required in practical applications. The net heat should be reported to the nearest 0.005 MJ/kg.

Note: Usually the gross heat of combustion is reported for fuel oils in preference to net heat of combustion to the nearest 0.005 MJ/kg.

To obtain the gross or net heat of combustion in cal (I.T.)/g or Btu/lb divide by the appropriate factor reporting to the nearest 0.5 cal/g or 1 Btu/lb.

$$Q_{Btu}/lb = (Q, MJ/kg)/0.002326 \qquad \text{(Eq. 8)}$$
$$Q_{cal}/g = (Q, MJ/kg)/0.0041868 \qquad \text{(Eq. 9)}$$

7.2 Forms

Figure 7-1 is a representative Heat of Combustion Pre-Test Checklist; Figure 7-2 is a representative Test Data Sheet, and Figure 7-3 is a typical calibration sheet.

Figure 7-1.
Pre-Test Checklist.

Heat of Combustion
Pre-Test Checklist

Request No. ______________

	Initial Yes	Initial No
1. Applicable ASTM standard reviewed?	☐	☐
2. OWI reviewed?	☐	☐
3. All safety equipment available and working properly?	☐	☐
4. Oxygen regulator operates properly?	☐	☐
5. Sample MSDS read?	☐	☐
6. Test plan read?	☐	☐
7. Calorimeter has sufficient water for testing?	☐	☐
8. Air leaks present?	☐	☐
9. Touch screen checked for error messages?	☐	☐
10. Benzoic acid standard within 1% of expected value?	☐	☐

Remarks/Discussion of Discrepancies:

____
____
____
____
____
____
____
____
____
____
____

______________________________ ______________________
Test Operator Date

x/06 EM10-F-CHM-093

Note: Representative form. Refer to Forms Master list for current version.

Figure 7-2.
Heat of Combustion Data Sheet.

Heat of Combustion Test Data Sheet

Project:_____________ ____ Test No.:_________
Request ID No.:__________ Requester:___________________________

Manufacturer:____
Composition:____
Specification:____

Sample Weight:___________ (g)
Temperature Change: _______(°C)
Heat of Combustion: __________(cal/g)
Fuse Wire Correction: __________(cal/g)
Energy Equivalent Factor: _________(cal/g)

Combustion Aid Used? Yes ☐ No ☐
Type of Combustion Aid __________________________
Weight of Combustion Aid _______________________ (g)
Combustion Aid Total Energy_____________________ (cal/g)

Special Sample Preparation Notes:

____
____
____
____
____

Other Remarks:

____
____
____
____
____

Test Operator: __________________________ Date: ________________

x/07 EM10-F-CHM-094

Note: Representative form. Refer to Forms Master list for current version.

Calibration Statement: Categories IV and V Equipment

Calibration is required before use per MPR-8730.5.

(Calibration before use for each test series and periodic testing by the Using LIne Organization)

Calibration Contacts: EM10/James Perkins, EM10/Mark Griffin

User Name:______________________________

Equipment Description:______________________________

(attach multiple components sheets if necessary)

Manufacturer:______________________________

ECN:__________ Serial No.:__________ Model No.:__________

Date of Calibration:______________________________

Type of Software and Version:______________________________

____

Listing of Standards Associated with Calibration:

____

____

____

____

____

Are standards National Institute of Standards and Technology (NIST) traceable? ☐ Y ☐ N

Did calibration meet equipment manufacturer's specifications? ☐ Y ☐ N

Calibration was performed by:______________________________

Remarks:

____

____

____

____

____

____

____

____

1/05 EM10-F-CHM-018

Note: Representative form. Refer to Forms Master list for current version.

Figure 7-3. Typical Calibration Statement.

7.3 Running a Spike (Combustion Aid) in the Bomb Calorimeter

7.3.1. **Perform** steps 4.5.10 through 4.5.22 to run the promoter (spike) in the calorimeter to determine its heat of combustion in units of cal/g. A spike could mineral oil, the benzoic acid standard, or another chemical whose properties are well understood.

7.3.2. Once the heat of combustion of the promoter has been determined, **set up** the machine to account for the added combustion of the promoter, as follows:

- **Go** to the **Main Menu** screen and into the **Operating Controls** subfolder. Then, **go** to the **Spiking Correction** subolder.
- In the **Spiking Correction** subfolder, **set** the **Use Spiking** option to the ON position.
- **Select** the **Heat of Combustion of Spike** folder, and **enter** the value of the spike calculated earlier in units of cal/g.
- **Set** the **Prompt for Spike before Weight** to the ON position.

7.3.2. **Return** to the **Main Menu**, and **run** test as normal.

7.4 Spiking Sample with Mineral Oil in the Bomb Calorimeter

To spike the sample with mineral oil, the procedure described in section 7.3 can be used; however, the sample also needs to be taken into consideration. The total energy output (sample and spike combined) should approximate the energy obtained when running a benzoic acid standard (6318 cal/g) by itself.

7.4.1. **Place** the sample cup into the scale and **tare** it.

7.4.2. **Add** the sample to the cup, and **record** its weight.

7.4.3. **Have** a rough idea of the caloric value of the sample before proceeding to the next step.

7.4.4. **Tare** the weight of the sample, add 0.1 to0.5 ml of mineral oil, and **record** the weight.

7.4.5. **Record** both the weight of the sample and the oil, which is the value that will be entered when running the sample/spike.

8.0 Records

8.0 Records

Records for Building 4623 shall consist of (a) memoranda that contain test results and that are stored electronically in the Materials and Processes Technical Information System (MAPTIS) and (b) calibration records.

8.1 Memoranda

Memoranda containing test results shall be retained indefinitely by EM10. These memoranda shall be stored electronically in the MAPTIS database and shall be accessible by test request number or memorandum number.

8.2 Calibration Records

8.2.1. All equipment requiring calibration shall be in current calibration, in accordance with EM10-OWI-CHM-050, *Building 4623 Guidelines for General Operations.*

8.2.2. Form EM10-F-CHM-018 (Figure 7-3, section 7) shall be used to document the calibration of all Category IV and Category V equipment.

8.3 Maintenance of Records

8.3.1. Memoranda less than 10 years old shall be maintained in ready-access files in MAPTIS; memoranda 10 years old or older shall be automatically transferred to historical files.

8.3.2. Calibration records shall be maintained on site for a minimum of 10 years, filed and indexed by test request number. These shall be stored in a manner that will protect them, *e.g.*, in a test folder stored in a metal file cabinet. After 10 years, calibration records shall be transferred to historical files.

8.3.3. The original test records shall be saved for a minimum of 5 years.

9.0 Tools, Equipment, and Materials

9.1 Standard Configuration of Parr Bomb Calorimeter

Heat of combustion is determined in this test method by burning a weighed sample in an oxygen bomb calorimeter under controlled conditions. The heat of combustion is computed from temperature observations before, during, and after combustion, with proper allowance for thermochemical and heat transfer corrections. Either isothermal or adiabatic calorimeter jackets can be used. The calorimeter consists of an oxygen bomb, a vessel with stirrer, and the water in which the bomb is immersed.

The model 6200 Calorimeter (Figures 9-1 and 9-2) is a true isoperibol calorimeter. A water jacket surrounds the combustion bomb and its “bucket.” A microprocessor-based controller monitors both the temperature of the bucket and the jacket and performs the necessary heat leak corrections that result from differences in these two temperatures. These corrections are applied continuously throughout a test rather than as a final correction based on pre- and post-test measurements. This method has basically replaced adiabatic calorimetry as the principal operating mode for routing calorimetric testing.

The Parr 1104 Oxygen Bomb (Figure 9-3) is designed for combustion tests of explosives and other fast-burning, high-energy samples that burn with extreme violence. The bomb is also recommended for use with materials whose combustion characteristics are unknown or unpredictable.

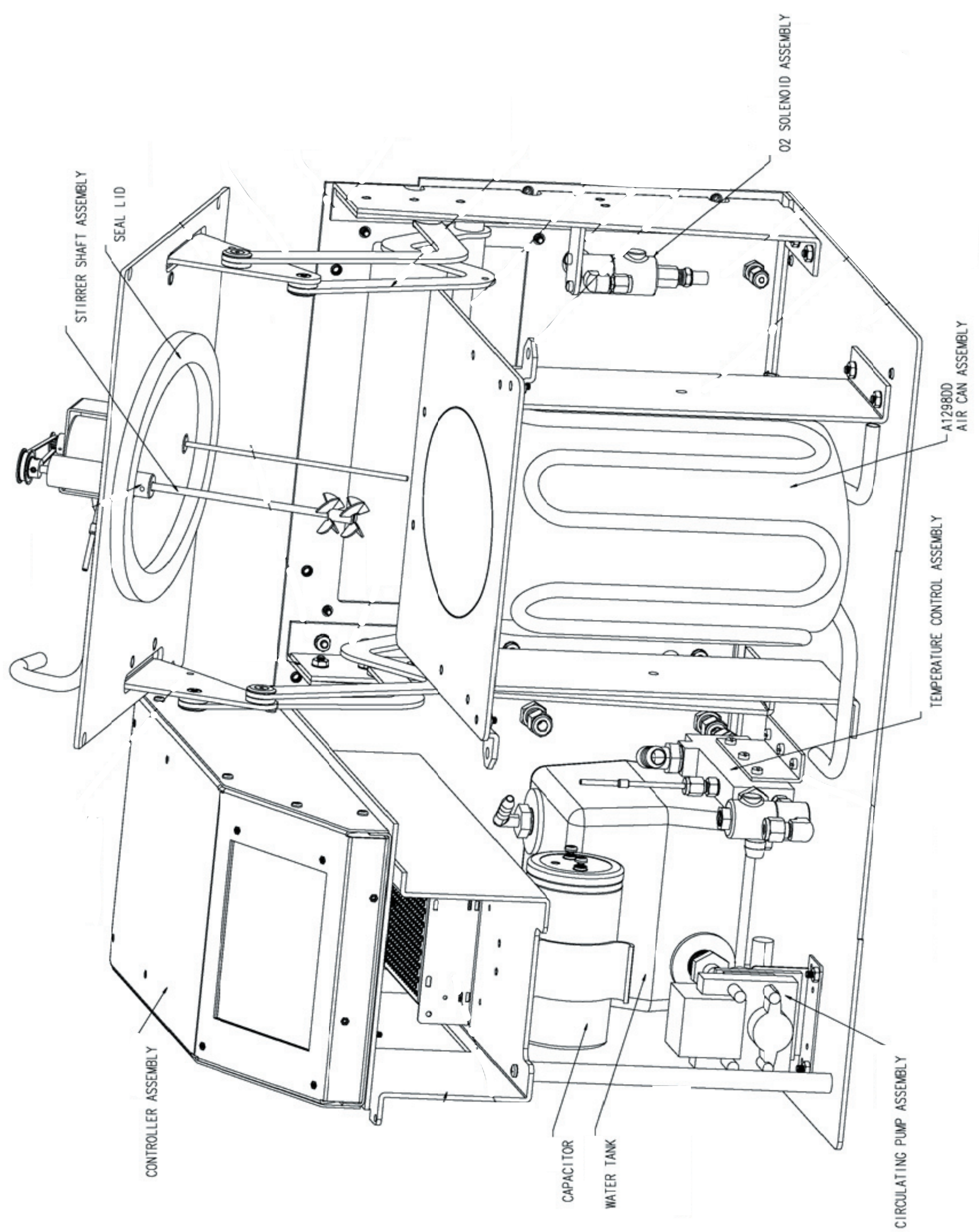
The 1104 is a heavy-walled, 240-ml vessel that handles samples liberating up to 12,000 calories using an oxygen charging pressure upto 45 atm. The sample is contained in a combustion cage designed to muffle the shock forces produced by explosive samples. The capsule is anchored to the cage so the explosion will not dislodge it. (See Figure 9-4.)

In some cases, it is not necessary to use the cage (Figure 9-5). It is strongly recommended that the user perform preliminary combustions with small samples, then increase the sample size and vary the oxygen pressure carefully to determine the optimum charge. In these cases, a lighter capsule may be substituted.

The bomb features an automatic inlet check valve and an adjustable needle valve for controlled release of residual gases following combustion. These are intended for samples ranging from 0.6 to 1.2 g.

The 6510 water handling system allows operation of the calorimeter with a closed-loop system. This system incorporates a precision pipette for measuring and delivering the water for the bucket at the same fixed temperature for each test. It also provides cooling water to the jacket of the 6200 calorimeter. The 6510 water handling system includes a thermo-electric cooler and does not require a separate cooler.

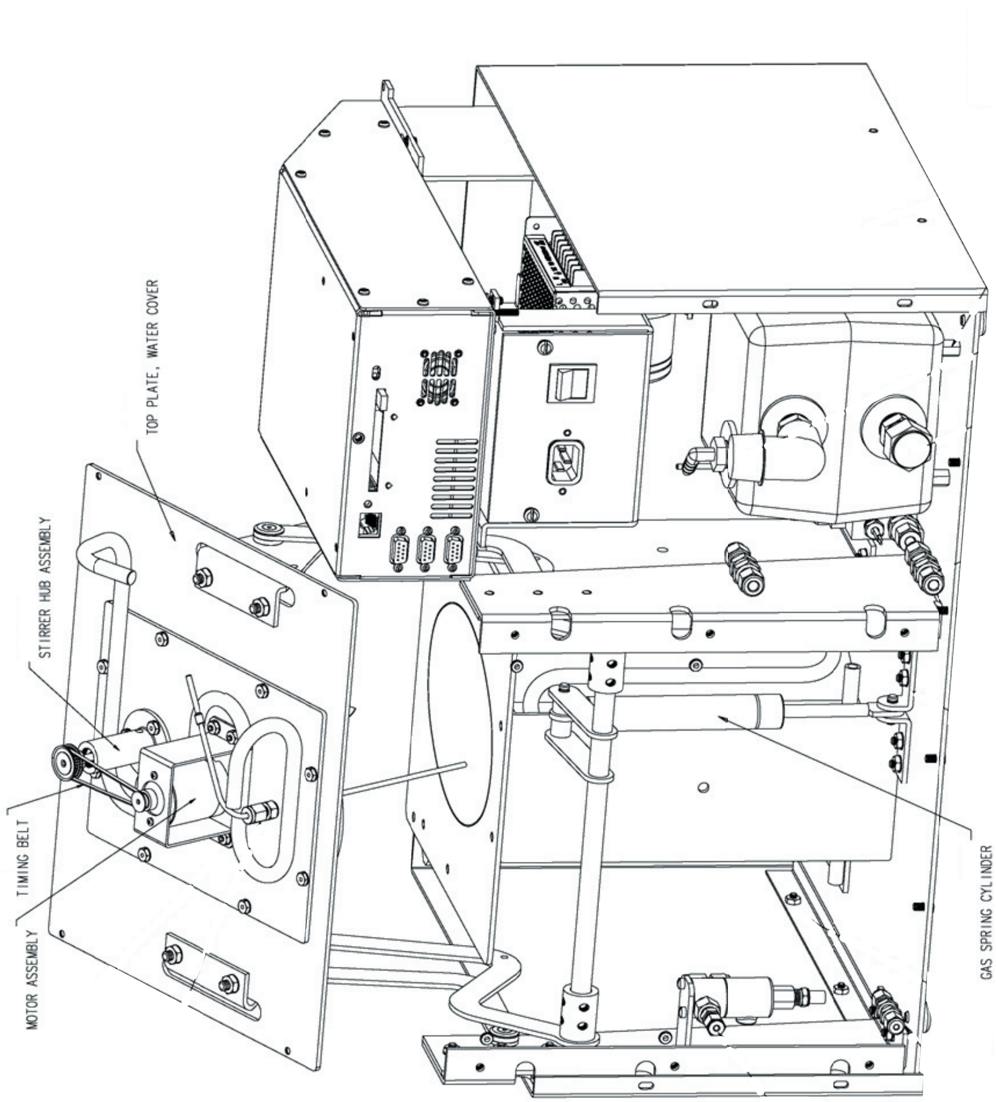
Figure 9-1.
Parr 6200 Bomb Calorimeter (cutaway, front) (Illustration adapted from Parr 6200 Calorimeter Operating Instruction Manual)



Note: Illustration is representative. Actual appearance may vary.

To speed and simplify the bomb filling connection, the 6200 calorimeter has a semiautomatic system for charging the bomb with oxygen. The oxygen is connected to a microprocessor-controlled solenoid installed in the calorimeter. Built-in safety provisions will prevent an accidental overcharge, and an error message will be shown *if the desired pressure is not obtained.*

Figure 9-2.
Parr 6200 Bomb Calorimeter (cutaway, rear) (Illustration adapted from Parr 6200 Calorimeter Operating Instruction Manual)



Note: Illustration is representative. Actual appearance may vary.

9.3 Procedure for Deviations

Not applicable.

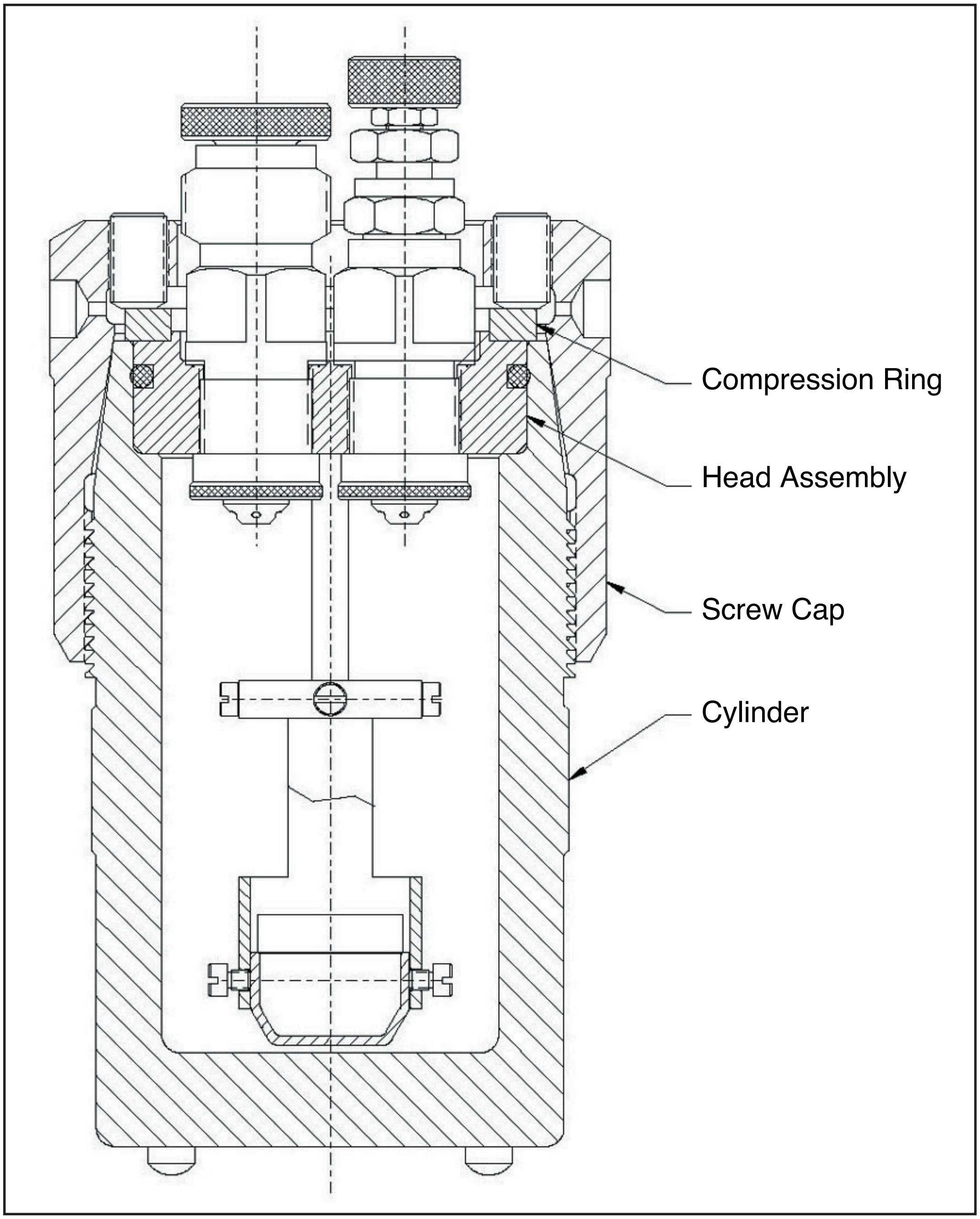


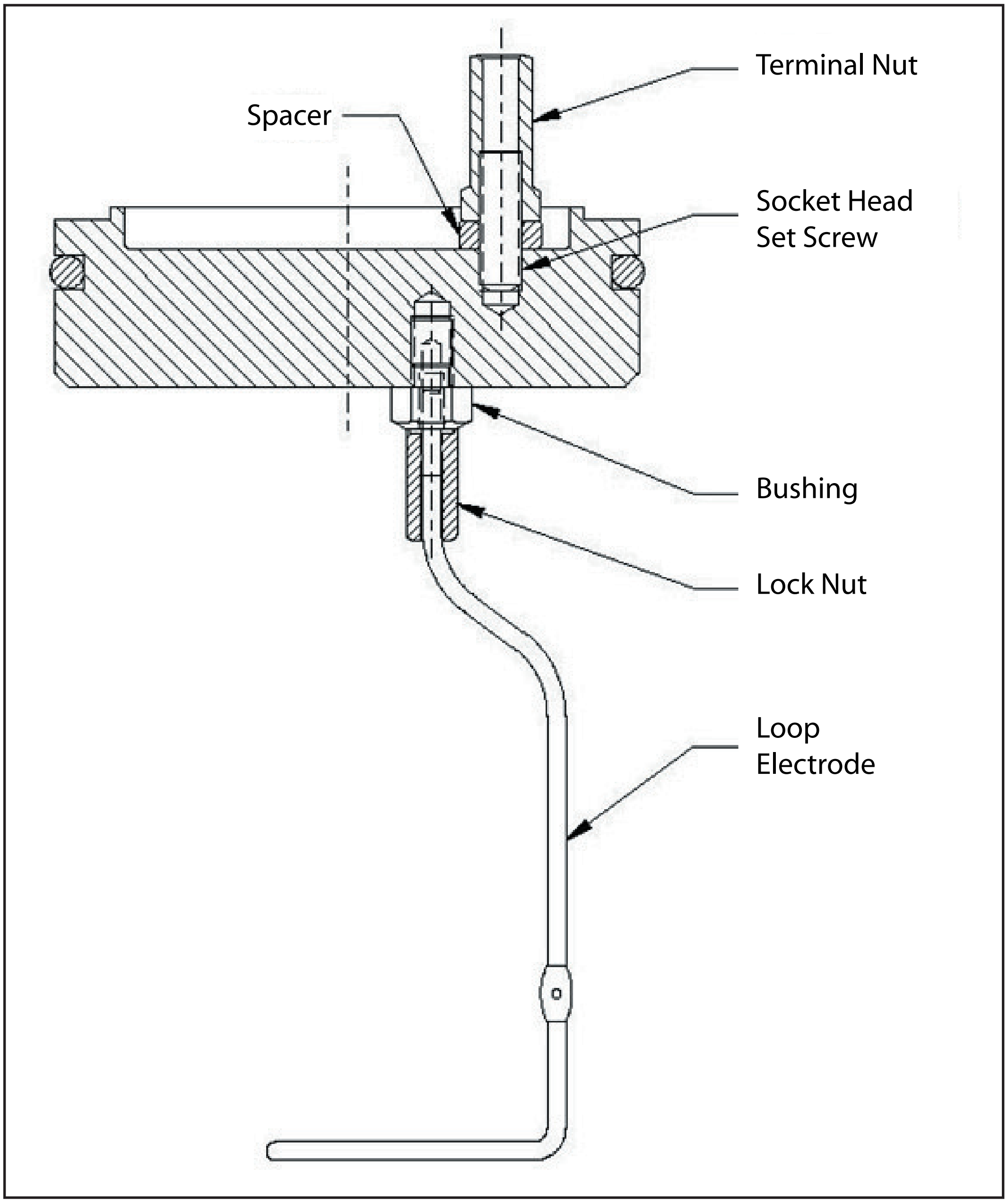
Figure 9-3.
1104 High-Pressure Oxygen Bomb

Note: Illustration is representative. Actual appearance may vary.

9.4 Tester Maintenance

The standard maintenance program for the Parr Bomb Calorimeter 6200 and related control equipment is divided into post-test and periodic service. In addition, the program involves a maintenance log, calibration, and a required spare parts inventory.

Figure 9-4.
Loop Electrode Test Configuration



Note: Illustration is representative. Actual appearance may vary.

9.4.1. Post-Test Maintenance: The chromium-plated, steel alloy screw cap on the bomb requires special care to keep the threads from rusting and to prevent seizure. Always **dry** the cap thoroughly after it has been used, and **store** the bomb with the screw cap removed. **Keep** a light coating of anti-seize lubricant on the threads but **do not use** this or any lubricant on any other parts of the bomb.

9.4.2. Periodic Maintenance:

9.4.2.1. During extend periods of inactivity (overnight or longer), **close** the oxygen tank valve to prevent leakage.

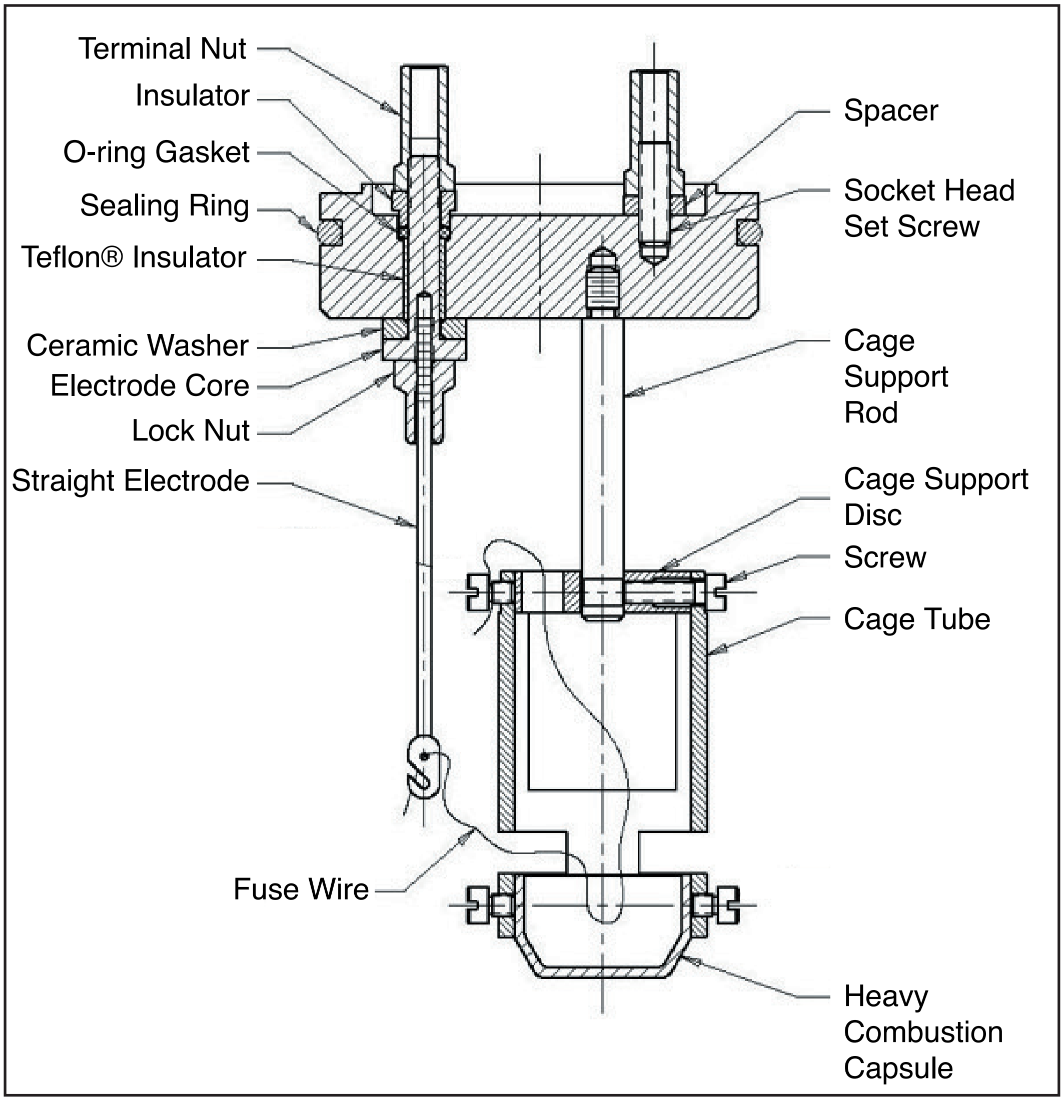


Figure 9-5. Combustion CageTest Configuration

Note: Illustration is representative. Actual appearance may vary.

9.4.2.2. When changing oxygen tanks, **close** the tank valve, and **push** the **O2 Fill** button to exhaust the system. **Do not use oil** or combustible lubricants on this filling system or on any devices handling oxygen under pressure. **Keep** all threads, fitting and gaskets clean and in good condition. **Replace** the two 394HCJE O-rings in the slip connector if the connector fails to maintain a tight seal on the bomb inlet valve.

9.4.2.3. Basic maintenance of the bomb includes replacing the O-rings, insulators, gaskets, and spacer after every 500 firings. *If the bomb is used for samples containing corrosive materials*, this maintenance shall be conducted be completed after 250 firings. Periodic cleaning may be performed on the exterior surfaces of the instrument with a damp cloth. **Disconnect** all power when cleaning the instrument.

9.4.2.4. A manufacturer's service of the oxygen bomb is recommended after

5000 firings. This service includes replacing all the seals; checking the dimensions of the cylinder, screw cap, and head; hydrostatically testing the seal; and proof firing the vessel using benzoic acid.

9.4.3. Maintenance Log. The test operator **shall document** any maintenance to the tester or setup in the Parr Bomb Calorimeter 6200 *Maintenance Log* to provide a history of the tester. Any deviation to standard maintenance shall be documented by the test operator and approved on the maintenance log by the responsible test engineer.

9.5 Calibration

Calibration of the Parr Bomb Calorimeter 6200 occurs periodically during system setup, as described in section 4.4.

9.6 Required Spare Parts Inventory

The test operator **shall verify** that the spare parts listed in Table 9-1 are available at the beginning of each test request, so that the testing of a material is completed as close to within 1 working day as possible.

Table 9-1.
Spare Parts Inventory

Part Description	Part Number	Quantity
Fuse Wire - Nickel Alloy	45C10	1 pack
Stainless Steel Capsules	43AS	6
Monel Washers	7VBCM	6
2-3/8” x 1/8” Buna O-rings	230A	6
NBR Gaskets	56A	6
3/16” x 1/16” Buna O-rings	238A	12
Sleeve Insulator	401A	2
Delrin Insulator	143AC	2
Lock Nuts	406A	2
Valve Seat - PCTFE	20VB	6
Sleeve Insulator	96AC	2
Copper Washer	109A	2
Lead Wire	A297E	2
STR Electrode	4A	2

10.0 Personnel Training and Certification

The nature of testing that occurs in the Building 4623 is complex and involves potential hazards; therefore, all Heat of Combustion test operators **shall be** Category 1 certified before conducting any test, and all Heat of Combustion tester maintenance personnel **shall be** Category 2 certified. This section describes the two levels of certification:

- **Category 1 Certification** qualifies personnel to perform basic test operations.
- **Category 2 Certification** qualifies personnel to maintain and modify testing apparatus.

Category 1 - Basic Operations

To be certified, all Heat of Combustion test operators must complete training in the following areas:

- Handling of Compressed Gas Cylinders
- Oxygen Compatibility
- General Safe Laboratory Practices
- High-Pressure System Safety.

Category 1 Certification also requires an annual physical examination conducted by the medical facility at Marshall Space Flight Center (or equivalent), including a hearing exam.

The operator **shall demonstrate** knowledge of the test and equipment by completing three successful test sets under the supervision of the test engineer. There is no emergency shutdown procedure for this tester.

Test operators **shall thoroughly read** the test OWI as part of the certification process. They **shall sign** a statement that they have read and understand the OWI and **shall be issued** personal copies of the OWI. The test engineer **shall give** the candidate a written test covering the OWI. A copy of this test, along with the signed statement and the training record, **shall constitute** verification of certification. Training records **shall be kept** on file as proof of training. These records **shall include** training expiration dates and required refresher courses.

These certifications **shall expire** after a period of 2 years. After that time, recertification **shall be** required.

Category 2 - Tester Maintenance and Modifications

Personnel seeking **Category 2 Certification** shall become qualified and certified in the following areas:

- *Compressed Gases and Working with Compressed Gas Lines and Fittings*
- *Basic Electrical Wiring.*

This qualification/certification **shall be achieved** through training classes approved by the candidate's supervisor or through training classes completed during previous employment.

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EMERGENCY PHONE NUMBERS

Emergency.......................... 911

Medical Center...................... 4-2390

Industrial Safety.................. 4-0046

Chemical Spills..................... 4-4357

Safety Monitor
Building 4623....................... 4-3571